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Associate Counsel (Patents)  
Code 10082  
Naval Research Laboratory  
Washington, DC 20375-5000

EXAMINER

SODERQUIST, ARLEN

ART UNIT	PAPER NUMBER
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1743

DATE MAILED: 06/02/2004

Please find below and/or attached an Office communication concerning this application or proceeding.

**Office Action Summary**

Application No.

09/544,344

Applicant(s)

SNOW ET AL.

Examiner

Arlen Soderquist

Art Unit

1743

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

**Period for Reply**

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If the period for reply specified above is less than thirty (30) days, a reply within the statutory minimum of thirty (30) days will be considered timely.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

**Status**

- 1) ☒ Responsive to communication(s) filed on 01 March 2004.
- 2a) ☒ This action is **FINAL**. 2b) ☐ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

**Disposition of Claims**

- 4) ☒ Claim(s) 1,2,4-9,21,22 and 24-39 is/are pending in the application.
- 4a) Of the above claim(s) 39 is/are withdrawn from consideration.
- 5) ☐ Claim(s) \_\_\_\_\_ is/are allowed.
- 6) ☒ Claim(s) 1,2,4-9,21,22 and 24-38 is/are rejected.
- 7) ☐ Claim(s) \_\_\_\_\_ is/are objected to.
- 8) ☐ Claim(s) \_\_\_\_\_ are subject to restriction and/or election requirement.

**Application Papers**

- 9) ☒ The specification is objected to by the Examiner.
- 10) ☒ The drawing(s) filed on 06 April 2000 is/are: a) ☒ accepted or b) ☐ objected to by the Examiner.
- Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).
- Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

**Priority under 35 U.S.C. § 119**

- 12) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☐ All b) ☐ Some \* c) ☐ None of:
1. ☐ Certified copies of the priority documents have been received.
  2. ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_\_.
  3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

\* See the attached detailed Office action for a list of the certified copies not received.

**Attachment(s)**

- 1) ☒ Notice of References Cited (PTO-892)
- 2) ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)
- 3) ☐ Information Disclosure Statement(s) (PTO-1449 or PTO/SB/08)  
Paper No(s)/Mail Date \_\_\_\_\_
- 4) ☐ Interview Summary (PTO-413)  
Paper No(s)/Mail Date. \_\_\_\_\_
- 5) ☐ Notice of Informal Patent Application (PTO-152)
- 6) ☐ Other: \_\_\_\_\_

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1. Newly submitted claim 39 is directed to an invention that is independent or distinct from the invention originally claimed for the following reasons: this application is a divisional application of the parent application and claim 39 is directed to subject matter claimed and now patented in the parent application.

Since applicant has received an action on the merits for the originally presented invention, this invention has been constructively elected by original presentation for prosecution on the merits. Accordingly, claim 39 is withdrawn from consideration as being directed to a non-elected invention. See 37 CFR 1.142(b) and MPEP § 821.03.

2. The following is a quotation of the first paragraph of 35 U.S.C. 112:

The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the same and shall set forth the best mode contemplated by the inventor of carrying out his invention.

3. Claims 5, 24, 26 and 37 are rejected under 35 U.S.C. 112, first paragraph, as failing to comply with the written description requirement. The claim(s) contains subject matter which was not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventor(s), at the time the application was filed, had possession of the claimed invention. Relative to claim 24, examiner was not able to find basis in the specification for the upper limitation of 2000 nm. Relative to claims 5 and 26 examiner was not able to find basis for the specificity that the various molecules have been defined. For primary aliphatic thiols examiner found basis for or specific examples of  $n = 3, 4, 6, 8, 10, 12, 16, 18$  and 20. For secondary and aliphatic thiols and heterofunctional araliphatic thiols examiner found no basis for the specificity claimed. For heterofunctional aliphatic thiols in addition to the values of  $n$  above examiner found basis for  $X = OH$ , halogen,  $COOH$  and  $NH_2$  ("and the like" in the listings on page 13 of the specification does not provide basis for the specificity that applicant has claimed). in claim 37, examiner cannot find basis for the "and combinations thereof" portion of the claim.

4. Claims 1-2, 4-9, 21-22, and 24-38 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention. In each of the independent claims applicant has included the language "and composed of an encapsulating monomolecular layer of ligand molecules each molecule having a single bonding site to a gold surface". For the following reasons this creates

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confusion in the claims. First, the claims have particles that have a core of a conductive metal or conductive metal alloy, but do not limit this to gold. Thus it is not clear if applicant intends to limit the particles to gold particles or alloys so that the molecules in the ligand shell can have a bonding cite to a gold surface or otherwise how the limitation is to be interpreted. If applicant intends to limit the metallic core to gold or a gold alloy then claim 2 and others with a similar limitation have a problem in that they do not further limit the independent claims. Second, in the heterofunctional molecules there are functional groups such  $\text{NH}_2$  that present a second gold surface bonding site and that portion of those claims fails to further limit the independent claims.

5. In the rejections that follow, examiner will be citing portions of the instant specification to show that various claimed properties are inherent in the teachings of the applied references. The format used will be bold italicized letters contained within parentheses.

6. The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless –

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

7. Claims 1-2, 4-5, 7-9, 21-22, 24-27 and 33-34 are rejected under 35 U.S.C. 102(b) as being anticipated by Bethell (*J. Electroanal. Chem.*). In the paper Bethell describes simple methods for the production of Au nanoparticles with narrow size distributions by reduction of tetrachloroaurate solutions in the presence of thiol-containing organic compounds which self-assemble on the Au surface. Stable solutions of somewhat larger particles can be produced if the thiol is absent. The thiol-derivatized materials are stable in air over long periods and can be handled in much the same way as simple organic compounds. Page 138, column 1, last full paragraph teaches nonanethiol derivatized particles having a core diameter of 1.5-3 nm. Subheading 2.1 contains a reference to an article by M. Brust which is found cited in the instant specification as one of the methods for making the metal core-ligand particles of the instant invention (*see page 13, line 23 to page 14, line 8*). In figure 1(b) a spectrum of dodecanethiol-derivatized particles is presented (*see examples 10-13, page 33 line 9 to page 34, line 10 for a description of particles made with dodecanethiol*). Table 1 teaches a range of dithiol containing molecules. Each of these is clearly within the thiol derivatives taught in the instant

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specification (*see page 12 line 21 to page 13 line 11 and 26-30 page 39, line 12 to page 41, line 23*). Using dithiols as the derivatizing spacer units, methods were developed for the preparation of materials in 3-dimensional form and as thin films attached to a solid substrate (figure 3, and the only full paragraph on page 139). (*A comparison of this procedure with that found in examples 26-30 shows that there is significant similarity and that the Bethell process forms a self-assembled layered structure as in the examples.*) Such materials show conductivities that mimic the behavior of semiconductors and that depend markedly on the structure of the dithiol used to link the Au particles together. Thus there is inherently a structure capable of being used to measure conductivity. The increase in conductivity with increasing temperature probably involves activated electron hopping from particle to particle. Surfaces treated with a coating of the materials show electroreflectance changes with applied potential that also differ according to the structure of the dithiol spacer. Unusual effects were observed on heterogeneous electron transfer from electrode surfaces treated with layers of the Au nanoparticles and dithiol spacers. Applications for these nanostructured materials can be envisaged, which range from submicroelectronic devices and circuitry to electrical modification of the reflectance of glass. Such applications will require a multidisciplinary approach with a substantial organic chemical research input. Section 4 and figure 5 discuss other methods of making the clusters which use multi-functional groups on the ligand layer surrounding the metal core. (*Additional sections of the instant specification that are relevant to the inherency of the Bethell structure are page 13, lines 16-18 teaching that the thickness of the ligand shell is determined by the size of the ligand molecule and page 24, lines 15-25 showing that ligand shell thickness on particles made with of alkanethiols having 6, 8, 12 or 16 carbons, C6, C8, C12 and C16, are 0.71 nm, 0.86 nm, 1.16 nm and 1.58 nm, respectively. This clearly shows that the thickness of the ligand shell of the particles is anticipated by the Bethell reference due to the use of similar compounds to synthesize the self assembled materials in a similar manner.*)

8. Claims 1-2, 4-5, 7-9, 21-22, 24-27 and 33-34 are rejected under 35 U.S.C. 102(b) as being anticipated by Anders. In the paper Andres teaches self-assembly of a two-dimensional superlattice of molecularly linked metal clusters. Close-packed planar arrays of nanometer-diameter gold clusters that are covalently linked to each other by rigid, double-ended organic molecules were self-assembled. Au nanocrystals, each encapsulated by a monolayer of alkyl

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thiol molecules were cast from a colloidal solution onto a flat substrate to form a close-packed cluster monolayer. Organic interconnects (aryl dithiols or aryl diisonitriles) displaced the alkyl thiol molecules and covalently linked adjacent clusters in the monolayer to form a two-dimensional superlattice of metal quantum dots coupled by uniform tunnel junctions. Electrical conductance through such a superlattice of 3.7-nm-diameter Au clusters, deposited on a SiO<sub>2</sub> substrate in the gap between two Au contacts and linked by an aryl di-isonitrile [1,4-di(4-isocyanophenylethynyl)-2-ethylbenene], exhibited nonlinear Coulomb charging behavior. Page 1692 in the two paragraphs that bridge the three columns, ligand shell thickness of ~1.2 nanometers are taught for the dodecanethiol ligand shell along with the possibility that the molecules on adjacent clusters interpenetrate to give a cluster spacing that is less than the expected layer thickness. For the dithiol linked clusters the spacing is about 1.7 nm between clusters using a 2 nm length molecule as the linking molecule. In the last full paragraph of the center column of page 1691, Andres teaches that the synthetic method used produces particles that are equivalent to those produced by the Brust method (see reference 5). The advantage of using the Andres method is flexibility in the formation of the particles. Since Andres teaches particles of the correct diameter having a ligand shell of the proper thickness and means for measuring the electrical conductivity of the material, the claim elements are met and there is clear anticipation of the claimed structure.

9. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103(a) are summarized as follows:

1. Determining the scope and contents of the prior art.
2. Ascertaining the differences between the prior art and the claims at issue.
3. Resolving the level of ordinary skill in the pertinent art.
4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

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10. Claims 2, 4, 6, and 22 are rejected under 35 U.S.C. 103(a) as being unpatentable over Bethell as applied to claims 1 and 22 above, and further in view of Natan (US 5,609,907). Bethell does not teach other types of metal colloids or the use of amine functions in the coating materials.

In the patent Natan teaches the formation of self-assembled metal colloid layers. Figure 1A with its associated discussion teach that the colloids can be gold, silver or other suitable metals. Column 3, lines 39-59 and the brief description of Figures 1A and 1B teach the additional use of amine and other functional groups in addition to thiols used to immobilize the colloids on a surface. The brief discussion of the figures also includes colloids having two layers of metal. Figure 1D shows the various levels of self-assembled colloids including multilayered (bulk).

It would have been obvious to one of ordinary skill in the art at the time the invention was made to incorporate the metal cores and metal interacting functional groups of Natan into the self-assembled colloid structures of Bethell because as shown by Natan the specifically claimed metals and functional groups would have been recognized as functional equivalents to those of Bethell relative to the formation of the self assembled colloid layers.

11. Claims 27 and 29-32 and are rejected under 35 U.S.C. 103(a) as being unpatentable over Bethell as applied to claims 1-2, 4-5, 7-9, 21-22, 24-26, and 33-34 above, and further in view of Terrill and Andres. Bethell does not teach the type of electrode used or the measurement equipment used.

In the paper Terrill discusses NMR, SAXS, Thermal, and Electron Hopping Studies of alkanethiol stabilized gold cluster monolayers in three dimensions. Au clusters stabilized by chemisorbed monolayers of octane-, dodecane-, or hexadecanethiolate were studied in solution and in the solid phase. These materials can be pumped free of solvent to form a dark brown solid that can be re-dissolved in nonpolar solvents. Their exceptional stability suggests that they may be viewed as cluster compounds. The self-assembled alkanethiolate monolayers stabilizing the metal clusters can be studied by using techniques that are insufficiently sensitive for study of a monolayer on a flat surface (e.g.,  $^1\text{H}$  and  $^{13}\text{C}$  NMR, elemental analysis, DSC, thermogravimetry (TGA), diffusion-ordered NMR spectroscopy (DOSY)). Results from such measurements (combined with SAXS data on solutions of the clusters and AFM and STM images) are

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consistent with a small, monodisperse (12 Å radius) Au core, which modeled as a sphere contains ~ 400 Au atoms and ~ 126 alkanethiolate chains, or if modeled as a cuboctahedral structure contains 309 Au atoms and ~ 95 alkanethiolate chains. High-resolution NMR spectra of cluster solutions display well-defined resonances except for methylenes nearest the Au interface; the absence of the latter resonance is attributed to a combination of broadening mechanisms based on the discontinuous change in magnetic susceptibility at the metal-hydrocarbon interface and residual dipolar interactions. Films of the dry, solid cluster compound on interdigitated array electrodes (see pages 12538-12539, experimental section for preparation and measurements) exhibit current-potential responses characteristic of electron hopping conductivity in which electrons tunnel from Au core to Au core. The electron hopping rate decreases and the activation barrier increases systematically at longer alkane chain length. The results are consistent with electron transport rate control being a combination of thermally activated electron transfer to create oppositely charged Au cores (cermet theory) and distance-dependent tunneling ( $\beta = 1.2 \text{ \AA}^{-1}$ ) through the oriented alkanethiolate layers separating them. See pages 12545 - 12548 for a discussion of the electrical measurements.

In the paper Andres teaches self-assembly of a two-dimensional superlattice of molecularly linked metal clusters. Close-packed planar arrays of nanometer-diameter gold clusters that are covalently linked to each other by rigid, double-ended organic molecules were self-assembled. Au nanocrystals, each encapsulated by a monolayer of alkyl thiol molecules were cast from a colloidal solution onto a flat substrate to form a close-packed cluster monolayer. Organic interconnects (aryl dithiols or aryl diisonitriles) displaced the alkyl thiol molecules and covalently linked adjacent clusters in the monolayer to form a two-dimensional superlattice of metal quantum dots coupled by uniform tunnel junctions. Electrical conductance through such a superlattice of 3.7-nm-diameter Au clusters, deposited on a  $\text{SiO}_2$  substrate in the gap between two Au contacts and linked by an aryl di-isonitrile [1,4-di(4-isocyanophenylethynyl)-2-ethylbenzene], exhibited nonlinear Coulomb charging behavior. Page 1692 in the two paragraphs that bridge the three columns, ligand shell thickness of ~1.2 nanometers are taught for the dodecanethiol ligand shell along with the possibility that the molecules on adjacent clusters interpenetrate to give a cluster spacing that is less than the



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expected layer thickness. For the dithiol linked clusters the spacing is about 1.7 nm between clusters using a 2 nm length molecule as the linking molecule. In the last full paragraph of the center column of page 1691, Andres teaches that the synthetic method used produces particles that are equivalent to those produced by the Brust method (see reference 5). The advantage of using the Andres method is flexibility in the formation of the particles.

It would have been obvious to one of ordinary skill in the art at the time the invention was made to incorporate the electrodes and measurement apparatus of Terrill into the formation method of Bethell because of their known use and sensitivity for measuring resistivity of self-assembled colloid clusters as shown by Terrill. It also would have been obvious to one of ordinary skill in the art at the time of the invention to use the ligand displacement method of forming the linked clusters in producing the layered structure of Bethell because of the greater flexibility in synthesis as taught by Andres

12. Applicant's arguments filed March 1, 2004 have been fully considered but they are not persuasive. Relative to the thickness of the coating in Bethell the reference is not required to be taught if it is an inherent property of the structure being taught. It should be clear that the thickness is an inherent property since Bethell uses the same materials as in the instant specification. (thiols with a carbon chain between 3 and 20 atoms long) to synthesize the particles by the same method as Bethell or a method which is equivalent to Bethell. The same is true if all of the claimed elements are present such as electrodes. The fact that the structure is not taught as being used for applicant's intended purpose does not in any way prevent the taught structure from being capable of use in the intended manner. In response to applicant's argument that the Bethell reference does not teach the structure used for sensing, a recitation of the intended use of the claimed invention must result in a structural difference between the claimed invention and the prior art in order to patentably distinguish the claimed invention from the prior art. If the prior art structure is capable of performing the intended use, then it meets the claim. In a claim drawn to a process of making, the intended use must result in a manipulative difference as compared to the prior art. See *In re Casey*, 152 USPQ 235 (CCPA 1967) and *In re Otto*, 136 USPQ 458, 459 (CCPA 1963). The Andres reference explicitly teaches that the coating of the particles formed is within the claimed thickness. And the above comments are also relevant to the Andres reference. Therefore there is no question that this reference anticipates the claims. In

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response to applicant's arguments, the recitation of a chemical species has not been given patentable weight because the recitation occurs in the preamble. A preamble is generally not accorded any patentable weight where it merely recites the purpose of a process or the intended use of a structure, and where the body of the claim does not depend on the preamble for completeness but, instead, the process steps or structural limitations are able to stand alone. See *In re Hirao*, 535 F.2d 67, 190 USPQ 15 (CCPA 1976) and *Kropa v. Robie*, 187 F.2d 150, 152, 88 USPQ 478, 481 (CCPA 1951). Relative to this examiner points out that the interaction with a chemical species is an inherent property of the material being used to interact with the species. Thus since the same materials are taught by Bethell and Andres as found in the instant specification, the body of the claim does not depend on the recitation of a chemical species for completeness and the limitations are able to stand alone. Applicant is directed to claim 1 relative to arguments about the sensor being an electrical chemical sensing device. Claim 1 is of a scope that encompasses much more than electrically sensing a chemical of interest. Relative to the recitation of a sensor, page 1, line 19 to page 17, line 7 teach both optical and electrical properties being measured. Furthermore page 19 lines 3-12 teach a battery and a current meter as the equipment necessary to measure (sense) the electrical conductivity to determine if change has occurred. Therefore this is the scope examined in the interpretation of the general recitation of a sensor as found in most of the claims.

The combination including the Natan reference is used to show that other types of ligands and metals can be used to form the particles. The obviousness is based on the similarity of the methods and the final products. The claims being rejected by the combination are generally the article of manufacture rather than the sensor claims. Thus the claims are directed toward an article that could have several uses such as an optical sensor. This is consistent with the instant specification as noted above. Thus the combination of Bethell with Natan is a proper combination showing the obviousness of substituting the materials of Bethell with other materials.

Relative to the combination including the Terrill reference applicant is first directed to claim 24 which has been rejected as introducing new matter into the claims. The prior claim 24 set a film thickness of 5 to 10,000 nm (0.005 – 10  $\mu$ m). Thus about half of the range of the film thickness taught by Terrill is within the specifically claimed thickness. However claim 24 is a

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dependent claim which does not limit the thickness of any claim that does not depend therefrom. With respect to all claims not having the thickness limitation, the argument is not commensurate in scope with the claims. Even with claim 24 it is not persuasive since there is a clear overlap in the ranges taught and claimed. Again relative to the method of forming the material through spraying, the claims are not limited to a material produced by that method and the argument is not commensurate in scope with the claims. Relative to the layering method, the Andres reference clearly shows the difference between the method of Bethell and the claimed method to be obvious because of the flexibility offered using a ligand displacement method.

13. Claims 35-36 are objected to as being dependent upon a rejected base claim, but would be allowable if rewritten to overcome the rejection(s) under 35 U.S.C. 112, second paragraph, set forth in this Office action and to include all of the limitations of the base claim and any intervening claims. The art of record fails to teach or suggest the claims combination or elements. It is noted that although the respective elements are conventional as evidenced by the page 21, line 17 to page 22, line 8, examiner was not able to determine if they are part of the measurement apparatus taught by Terrill.

14. Applicant's amendment necessitated the new ground(s) of rejection presented in this Office action. Accordingly, **THIS ACTION IS MADE FINAL**. See MPEP § 706.07(a). Applicant is reminded of the extension of time policy as set forth in 37 CFR 1.136(a).

A shortened statutory period for reply to this final action is set to expire **THREE MONTHS** from the mailing date of this action. In the event a first reply is filed within **TWO MONTHS** of the mailing date of this final action and the advisory action is not mailed until after the end of the **THREE-MONTH** shortened statutory period, then the shortened statutory period will expire on the date the advisory action is mailed, and any extension fee pursuant to 37 CFR 1.136(a) will be calculated from the mailing date of the advisory action. In no event, however, will the statutory period for reply expire later than **SIX MONTHS** from the date of this final action.


15. The prior art made of record and not relied upon is considered pertinent to applicant's disclosure. The additionally cited art relates to sensor structures and nanoparticles having an organic ligand coating.

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Any inquiry concerning this communication or earlier communications from the examiner should be directed to Arlen Soderquist whose current telephone number is (571) 272-1265 as a result of the examiner moving to the new USPTO location. The examiner's schedule is variable between the hours of about 5:30 AM to about 5:00 PM on Monday through Thursday and alternate Fridays.

A general phone number for the organization to which this application is assigned is (571) 272-1700. The fax phone number to file official papers for this application or proceeding is (703) 872-9306.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).

A handwritten signature in cursive script, reading "Arlen Soderquist".

May 28, 2004

ARLEN SODERQUIST  
PRIMARY EXAMINER